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MINES BRANCH INVESTIGATION REPORT IR 58-180

EXAMINATION OF SCALE DEPOSIT ON TUNGSTEN ALLOY BLOCK

by

J. J. SEBISTY

PHYSICAL METALLURGY DIVISION

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SUMMARY OF RESULTS

Tests carried out to establish the nature and cause of heavy scaling of a tungsten alloy insert indicated that severe oxidation had occurred and this had been caused by firing of the investment mould at a high temperature with the tungsten insert in place in the mould.

It is not considered that material quality was a factor contributing to oxidation of the alloy.

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INTRODUCTION

On September 10th, 1958, a radiation shield of a type used in a cobalt 60 Beam Therapy Unit was received from Mr. Ridgeway, Atomic Energy of Canada Limited, Commercial Products Division, Ottawa. The part consisted of a composite tungsten block against which a thick-walled hollow brass container has been cast. The block actually consisted of three individual compacts which had been brazed together.

At some stage in the investment casting process used, a heavy scale deposit had formed on all surfaces of the block. Since a number of such shields had been similarly affected, making them useless for the intended application, examination of the surface deposit was requested by Mr. Ridgeway in order to establish its identity and factors responsible for its formation.

IDENTIFICATION OF DEPOSIT

X-ray diffraction examination of deposit samples removed from the exposed surface of the composite block and the interface between the block and brass container revealed that tungsten trioxide was a major constituent. An additional complex system of lines of fairly strong intensity were present in the patterns but these could not be identified by comparison with standard patterns available.

Similar examination of some white investment material adhering to the brass casting indicated this was essentially silica. An unidentified minor constituent was also noted.

SPECTROGRAPHIC ANALYSIS

Qualitative analysis of the deposit and investment material gave the results tabulated below. The presence of significant quantities

of copper and nickel in the deposit suggests that the material in the block is a ternary alloy of the type commonly used as shielding in radium beam therapy. The composition usually employed is 2 to 4% copper and 4 to 6% nickel. The unidentified diffraction patterns from the deposit are therefore probably related to compounds of copper, nickel and tungsten in the alloy.

The presence of silicon as the primary constituent of the investment material was confirmed by this analysis.

TABLE I
Qualitative Spectrographic Analysis*

	W	Ni	Cu	Zn	Si	Pb	Fe	Al	Ag
Deposit from side of block	1	1	2	4	3	5	3	3	3
Deposit from interface	1	1	2	3	3	4	3	4	3
Investment material	2	3	3	2	1	3	4	3	3

* - 1. Major Constituent

2. Minor Constituent

3. Strong trace

Cd, Ti, Mg, Cr, Sn, B, V, Mn - not detected or present in trace amounts only.

MICROEXAMINATION

To avoid cutting or otherwise damaging the tungsten block, Mr. Ridgeway provided a small sample from a second block for micro-examination. The latter was taken from the same shipment as the original and had suffered similar damage during the casting process. Examination of a piece from this sample after polishing and etching

revealed a structure consisting of large rounded grains of tungsten embedded in a continuous matrix of the Cu-Ni binder phase. This is typical of properly sintered material produced by the powder metallurgy technique. Some fine porosity was evident but the amount was not abnormal.

HEATING TESTS

Since the tungsten deposit was clearly associated with high temperature oxidation, various heating tests were carried out with the remainder of the above sample, to see if the deposit could be reproduced. Temperatures between 400°-900°C (752°-1652°F) were used. At the lower end of this range a superficial black oxide was formed for exposures of up to 2½ hr. At 700°C (1292°F) the oxide was bright yellow in color but even after 4 hr the deposit was relatively thin. Oxidation at 900°C (1652°F) was much more rapid and a powdery yellowish-black deposit showing most pronounced growth around the edges of the sample was formed after 1 hr. On the flat faces the scale was thin and adherent. This was colored black on the outside but underneath this outer layer, the characteristic dull yellow color, similar to that found on the original block, was revealed.

DISCUSSION

The examination and tests carried out established that the tungsten alloy block had been subjected to severe oxidizing conditions. A high temperature in the neighbourhood of 1000°C (1832°F) and an exposure of some length must have been used to account for the thickness and coherent nature of the scale.

Such oxidation could not have occurred in the casting process

and it can only be concluded that the block was in place in the investment mould when the latter was fired. This is supported by the more or less uniform thickness of oxide over the whole of the composite block including the curved top surface adjacent to the brass casting. In addition, there is the fact that the relative dimensions of block and casting did not conform. This was particularly evident along one face near a corner of the block where the oxide layer extended more than $1/32$ in. farther out than the corresponding brass casting surface. Such differences could not have been readily accommodated in final assembly of a fired investment mould. It is therefore apparent that the tungsten alloy block was inserted prior to firing and that conditions during this process were such as to cause severe deterioration of the tungsten alloy.

Mr. Ridgeway had stated that previous stocks of the same alloy had given no trouble during the casting process. In view of this and the apparently good metallurgical quality of the additional sample which was examined, it is not likely that inferior material quality was a factor contributing to the severe oxidation of the block in question but rather that the castings had been made by a different technique as outlined above.

CONCLUSIONS

Exposure of the tungsten alloy block to severe oxidizing conditions was responsible for formation of the surface deposit. The high temperature and lengthy period of exposure which would have been required to form such a heavy deposit suggests that the composite block must have been in place in the investment mould during firing of the latter.

It is not suspected that material quality was such as to have had any marked influence on the failure, and it is suggested that previous castings which were satisfactory had been made by inserting the tungsten alloy block into a pre-fired investment mould just prior to pouring.

JJS/sws